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Evaluation of aroma profile differences between *sué*, sautéed, and pan-fried onions using an innovative olfactometric approach

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Abstract

Background: Onions (*Allium cepa*) are widely used as a flavor agent ingredient in culinary preparations to bring specific cooked onion notes. In this study, three traditional types of preparations—*sué*, sautéed, and pan-fried onions—were used to investigate their differences in aroma profile.

Results: Headspace solid phase micro-extraction (HS-SPME) and gas chromatography (GC) coupled with mass spectrometry (MS), flame ionization detection (FID), and olfactometry were used to analyze the onion preparations. The study enables to identify 66 major compounds in the preparations. Among these compounds, sulfur compounds, aldehydes, and furans were the most represented. The pan-fried onion preparation distinguishes itself by the highest number of compounds represented in a large amount. This result is consistent with this mode of cooking that combines high temperature with long cooking time and favors the formation of compounds from the Maillard reaction and lipid oxidation. In comparison, the *sué* and sautéed preparations contain globally fewer compounds and, for most of them, in a lower amount compared to the pan-fried preparation. An innovative olfactometric approach was performed, based on a laboratory-developed software using an aroma wheel especially designed for the study of cooked onion. It enables an intuitive, efficient, and precise characterization of odor events along elution. A statistical comparison of intensities perceived for each odor detected during olfactometric analysis was used to understand the aroma balance and nuances perceived for these three traditional onion preparations. In accordance with chromatographic results, the pan-fried onion displays the highest number of odorant zones (65) associated with higher intensity scores and notably, to an enhanced perception of some Maillard compounds. *Sué* and sautéed onion profiles show an analog number of odorant zones (50 and 53), but the sautéed onion displays higher intensity scores and a particular contribution from pyrazines.

Conclusions: The olfactometric approach used completes advantageously the instrumental characterization of cooked onions samples obtained by these three traditional cooking processes and reveals the essential contribution of minor compounds to the aroma of cooked onions. Particular compounds and balanced profile intensities were pointed out to explain the specific aroma nuances of traditional *sué*, sautéed, and pan-fried onions.

Keywords: Cooked onion, Aroma profile, Olfactometric software, Sulfur compounds, Odorant compounds

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Background

Raw and cooked onions (*Allium cepa*) are traditionally used as a flavor agent ingredient in culinary preparations in order to spice dishes. They can bring global onion aroma to food as well as more specific cooked or caramelized notes depending on the techniques used in their preparation. The aroma profile of raw onions is dominated by sulfur-containing compounds. Among them, alk(en)yl-thiosulfinates bring a sharp pungent note to freshly cut onion [1]. These compounds are produced when onion cells are mechanically damaged, for example, by cutting, crushing, chewing, or by maceration. These processes enable an enzyme, the aliinase, initially present in the vacuole, to cut a non-volatile precursor found in cytoplasm, the S-alk(en)yl-L-cysteine-S-oxide, forming sulfenic acids and then thiosulfinates. The latter molecules are very unstable and give rise to further rearrangements leading to a wide variety of odorant compounds, such as mono-, di-, and tri-sulfur compounds, which can have a powerful sulfur or distinctive "freshly cut onions" odor [2]. Thiopropanal-S-oxide, also called the lachrymatory factor and responsible for the eye-stinging sensation, is also produced from S-alk(en)yl-L-cysteine-S-oxide precursor. This enzymatic reaction has been studied extensively over several years underlining the complex evolution of the volatile composition of raw onions [3].

Onions are often heated either for conservation or to create culinary notes. Heating processes applied to vegetables give rise to a complex evolution of the product that invariably produces additional volatile compounds through the autoxidation of some components, the thermal decomposition of others, and the initiation of Maillard-type compounds between amino acids and reducing sugars [1]. Some publications deal with the effect of cooking such as boiling, baking, frying, sterilization, and microwave treatments on the onion gas chromatography (GC) profile. As reported in these publications, the major compounds found in heated onions remain sulfur compounds such as mono-, di, tri-, tetrasulfides, thiophenes, and thiols but aldehydes, carboxylic acids, ketones, hydrocarbons, furans, pyrroles, and alcohols were also identified [4–8]. However, the contribution of these compounds in the perceived aroma of cooked onions was less investigated. Some polysulfides like propyl- and propenyl-containing di- and tri-sulfides were found to impact the aroma of cooked onions [9, 10]. 1-Propanethiol was also reported to contribute to the sweetness of cooked onion [11] as well as aldehydes seeming to bring a fried note to onions fried in corn oil or roasted in butter [1, 7]. However, many questions still remain to investigate. For example, if dimethylthiophenes are recognized to primarily bring the "fried onion note" in roasted onion in a study [9], another one disputes this claim [12]. The knowledge about aroma of cooked onions comes from older literature, and it is mainly based on model systems. Compounds' contribution to onion aroma was extrapolated from odor thresholds and descriptions of pure compounds in water [9, 10]. This knowledge should be improved through recent methodologies for extraction and GC analysis. More recently, the aroma of cooked onions and the contribution of oxygenated compounds to its "sweet flavor" onion were investigated by the use of GC coupled with olfactometry [8, 13]. However, no information was given concerning neither the intensity nor the number of detections for each odor perceived so that the respective contributions of the corresponding compounds were impossible to evaluate.

Olfactometry is a valuable methodology commonly used to investigate odorant active compounds in food aroma products and notably those with complex aroma profiles such as coffees, wines, or cheeses [14–16]. Considering the number of studies using this technique, little deals with the improvement of olfactometric data acquisition, although it is essential for the quality of the results. Acquisition systems found in bibliography are listed in Table 1. Despite inherent drawbacks, some olfactometric studies are still conducted with an oral transmission of judges' sensory impressions [17]. This practice leads to many restraints and bias such as perturbation of breathing rhythm, breakdown of sensorial perceptions, and generally mobilizes an operator to capture the judges' comments. This method can delay or complicate the recording of judge's perception leading, for example, to a possible failure to detect odorant compounds closely eluted like isomers. It also implies to restrict olfactometry sessions to a single judge which is time-consuming [18]. To avoid some of these biases, instrumental devices were developed to acquire automatically judges' perceptions. A pushing button was employed to record time and duration of an odor event [19–24]. Finger-span systems were also developed to record the intensity perceived for an odor by using the distance between the thumb and the major finger of the judge as a representation of the odor intensity score [25–27]. These tools are generally coupled with computer programs which gather duration and/or intensity data for further processing but, when they were recorded, descriptors were in most cases independently captured. Tape and digital recorders could be used to overcome the presence of an operator to record these data [19, 23, 25, 28]. They could be associated with devices such as Nose to Text software (Brechbuhler, Switzerland) or Voice Chromatogram Interface (Atas/GL Sciences, The Netherland, [29]) that merge vocal information into numerical data through a voice recognition system. However, these last appliances do not prevent perturbations associated with speaking. Besides, descriptors are often freely chosen by judges and for

Table 1 Comparative table of current acquisition devices used to record olfactometric data

Systems of acquisition	Recording odor events parameters			Respect of breathing rhythm and continuity of sensorial perceptions
	Time/duration	Intensity	Descriptor	
Internal acquisition software coupled with an tape recorder [23]	✓ Pushing button	x	✓ Oral recording	x
Coconut acquisition software (R. Almanza and P. Mielle, INRA dijon) coupled to an oral recorder [20]	✓ Keyboard	x	✓ Oral recording	x
Maestro software (Chrompack, Middelburg, The Netherlands) coupled with an oral recorder [21]	✓ Pushing button	x	✓ Oral recording	x
Internal acquisition software (using Pascal language) [24]	✓ Keyboard	✓	x	?
Internal acquisition software using C++ language [32]	✓ Icons at screen	x	✓ Icons at screen	✓
Internal acquisition program coupled with finger-span device (Almanza and Mielle, LRSA INRA Dijon 1990) [25, 26]	✓ Finger span	✓	x	✓
Internal acquisition program coupled with a finger-span system developed by SCL Ltd. (Dunedin, New Zealand) [27]	✓ Modified rotating finger span	✓	x	✓
AcquiSniff® (Biosens, France) coupled with a digital recorder [19]	✓ Pushing button	✓ Oral recording	✓ Oral recording	x
Sniffer 9000 (Brechbuller, Switzerland)	✓ Finger span	✓ Finger span	✓ Oral recording—Nose to Text conversion	x
Voice Chromatogram Interface (ATAS/GL Sciences, The Netherland) [29]	✓ Mouse button	✓ Mouse button	✓ Oral recording—conversion to text	x
Oniris software	✓ Aroma wheel interface with continuous intensity scale	✓	✓	✓

a same odor event, a consensus is complicated to obtain between judges. This lack of consensus is due to the difficulty for humans' brain to link the olfactory and the semantic memories and thus, to clearly associate a word to an odor. It explains why odor identification frequently leads to a fail [30]. To come through this problem, judges can be constrained to choose a term among a suitable short list [31]. One internal acquisition software experimented this methodology by asking a judge to choose an odor category among a short list preliminarily picked and then, on a second step, to precise their choice by clicking on a more precise descriptor [32]. Despite the intuitiveness of this software induced by the use of pictures, the description of the odor is made in several steps which delay data recording and could lead to a failure to characterize closely eluted odorants. Currently, and according to literature, there is no device that, all at once, prevents judges from speaking, makes them possible to record simultaneously duration time, intensity, and descriptor for each odor event, and enables a direct processing of the data collected.

Hence, the objective of this study was to compare aroma profiles of three traditional preparations of onions: *sué*, sautéed, and pan-fried onions, which are of a great industrial interest but poorly documented. Gas chromatography coupled with mass spectrometry, flame ionization detection, and olfactometry were used to analyze the volatile content resulting from these three modes of cooking. An innovative olfactometric approach based on a laboratory-developed device and a statistical comparison of odors intensity score was implemented to ensure an efficient comprehension of aroma nuances perceived for these traditional onion-based preparations.

Results and discussion

Chromatographic profiles of the headspace of *sué*, sautéed, and pan-fried onions

The present focus deals with the 66 identified compounds detected by flame ionization detection (FID) in the headspace of at least one of the three onion preparations (Table 2).

The sum of the unknown compounds' FID peak areas is less than 9 % in each preparation and is related to minor peaks. Identified compounds belong to various chemical families: 1 alcohol, 18 aldehydes, 3 carboxylic acids, 7 furans, 7 hydrocarbons, 6 ketones, 1 pyrrole, and 23 sulfur compounds. Most compounds are detected in the three preparations, but their abundance in terms of FID peak area is different. For 58 out of the 66 identified compounds, FID peak areas differ significantly between at least two modes of cooking ($p \leq 0.05$). The total peak area (TPA) detected in the pan-fried preparation is more than four times higher and 2.5 higher compared to the TPA detected in the *sué* and sautéed preparations, respectively, reflecting a greater quantity of volatile compounds generated in onions by pan frying. Forty compounds are detected in a significantly greater amount in the pan-fried preparation compared to both *sué* and sautéed ones confirming a real distinction of the pan-fried preparation. In addition, 26 compounds are found in significantly different amounts between *sué* and sautéed preparations showing a difference between these two modes of cooking.

Sulfur compounds are one of the most represented chemical families in the three onion preparations. Most of them were previously reported in raw [6, 33–36] and/or heated onions [4–8, 13]. The TPA of sulfur compounds detected is between three and five times higher in the pan-fried onion preparation than in sautéed and *sué* onion preparations, respectively. According to the analysis of variance (ANOVA) carried out on peak areas of each of these compounds, their amount is, for most of them, significantly higher in the headspace of pan-fried onion preparation than in the two other preparations.

Among sulfur compounds, 2,4-dimethylthiophene is one of the most detected compounds in the headspace of the samples (between 6.2 and 11.3 % of the TPA). This compound, as well as 2,5- and 3,4-dimethylthiophenes detected to a lesser extent, increases slightly from *sué* to sautéed preparations and then, substantially from sautéed to pan-fried ones. They may originate from the thermal conversion of propenyl methyl disulfides and propenyl propyl disulfides [9] or from a Maillard system involving cysteine and ribose [37]. 2- and 3-Methylthiophenes were only detected in pan-fried onion. These compounds were found in some processed vegetal material like roasted sesame seeds [38], but according to our knowledge, they have never been reported in neither raw nor cooked onions. Their origin in onion is unclear, but previous studies led in model media suggest that they could issue from heated cysteine or thiamine [39–41] or from a Maillard system involving cysteine and ribose [37].

Alk(en)yl mono-, di-, and tri-sulfides were detected in large numbers in the headspace of each sample and were all previously identified in raw [6, 33–36, 42] and/or cooked onions [4–8, 13]. These compounds were previously found to be the major volatile components in onion oil obtained by steam distillation [9, 10]. (Z)- and (E)-1-Propenyl methyl- and (Z)- and (E)-1-propenyl propyl-disulfides are among the most detected sulfides in the three preparations (between 1.3 and 12.2 % of the TPA). Dimethyl sulfide, (Z) and (E)-1-propenyl methyl sulfide, dimethyl disulfide, methyl propyl disulfide, (Z) and (E)-1-propenyl methyl disulfide, and dimethyl trisulfide are detected in significantly larger amounts in the pan-fried onion preparation compared to the other ones. Conversely, dipropyl disulfide, dipropyl trisulfide, and (Z)-1-propenyl propyl disulfide are in greater quantity in the headspace of *sué* onions than in sautéed and pan-fried ones. (E)-1-Propenyl propyl disulfide and methyl propyl trisulfide are detected in similar amounts in the three preparations. Alk(en)yl mono-, di-, and tri-sulfides originated from a series of reactions involving, in its first stage, the enzymatic reaction of aliinase on S-alk(en)yl-L-cysteine-S-oxide giving consecutively sulfenic acids, thiosulfinates, and finally mono- and polysulfides [2]. As mentioned above, some of these compounds can be further involved in the formation of dimethylthiophenes [9] which could be an hypothesis to explain their respective amount in the three types of preparations.

Three thiols: methanethiol, 1-propanethiol, and 1-pentanethiol, were identified in each sample (<0.5 % of the TPA). Their detected amounts increase from *sué* to sautéed onions and to pan-fried onions. Alkanethiols could be derived from interactions of Maillard reactions and lipids [37]. Methanethiol and propanethiol were previously found in raw [6, 34, 42] and cooked onions [4–8, 13], but to our knowledge, pentanethiol has never been reported in onions. However, this compound has already been identified in the volatile composition of chive (*Allium schoenoprasum* L.) [38]. Methanethiol is known to come from the degradation of methionine during Strecker reaction [43].

Methional was only detected in the headspace of the pan-fried onion preparation (0.1 % of the TPA). It is also known to come from degradation of methionine during Strecker reaction [43].

One trithiolane: 3,5-diethyl-1,2,4-trithiolane, was also detected in the samples. Its amount increased from the *sué* to the sautéed preparation and from the sautéed to the pan-fried preparation. This compound can be

Table 2 Volatile compounds identified by GC-MS-FID in *sués*, sautéed, and pan-fried onions

							Sué onion		Sautéed onion juice		Pan-fried onion juice		
	Compounds	CAS number			Calculated LRI	Literature LRI	Peak surface	Percent total peak area	Peak surface	Percent total peak area	Peak surface	Percent total peak area	Significance peak area
	Total peak area						2.4E+07^{c}		3.8E+07^{b}		1.0E+08^{a}		**
Carboxylic acid													
1	Acetic acid	64	19	7	1471	1427-1479	ND^{b}		ND^{b}		6.8E+05^{a}		***
2	Propanoic acid	79	9	4	1565	1487-1574	8.9E+04^{ab}		6.6E+04^{b}		1.3E+05^{a}		*
3	Hexanoic acid	142	62	1	1908	1815-1938	2.4E+05^{b}		2.6E+05^{b}		4.1E+05^{a}		*
	Total carboxylic acid						3.3E+05^{b}	1.4	3.3E+05^{b}	0.9	1.2E+06^{a}	1.2	***
Aldehydes													
4	Acetaldehyde	75	7	0	707	677-744	2.5E+05^{b}		5.3E+05^{b}		2.4E+06^{a}		***
5	Propanal	123	38	6	801	769-828	2.5E+06^{b}		1.6E+06^{c}		7.0E+06^{a}		***
6	2-Methylpropanal	78	84	2	819	800-858	ND^{b}		2.0E+05^{b}		2.1E+06^{a}		***
7	(E)-2-Propenal	107	2	8	852	828-864	9.2E+04^{b}		6.3E+04^{b}		1.5E+05^{a}		***
8	Butanal	123	72	8	884	850-911	5.1E+04^{b}		4.1E+04^{b}		1.6E+05^{a}		**
9	2-Methylbutanal	96	17	3	923	880-937	5.4E+04^{b}		9.3E+05^{b}		5.8E+06^{a}		***
10	3-Methylbutanal	590	86	3	926	902-961	1.4E+05^{b}		2.1E+06^{b}		1.6E+07^{a}		***
11	Pentanal	110	62	3	988	950-1003	4.1E+05^{b}		4.6E+05^{b}		6.9E+05^{a}		**
12	Hexanal	66	25	1	1097	1067-1099	1.5E+06^{c}		2.1E+06^{b}		2.8E+06^{a}		***
13	(E)-2-Methyl-2-butenal	497	3	0	1115	1069-1113	4.8E+04^{b}		6.5E+04^{b}		1.7E+06^{a}		***
14	(E)-2-Methyl-2-pentenal	623	36	9	1180	1143-1177	4.1E+05^{b}		2.9E+05^{b}		1.7E+06^{a}		***
15	Heptanal	111	71	7	1198	1174-1214	1.8E+05^{b}		2.7E+05^{a}		1.6E+05^{b}		*
16	Octanal	124	13	0	1306	1274-1340	1.4E+05^{c}		2.7E+05^{b}		4.7E+05^{a}		***
17	(E)-2-Heptenal	18829	55	5	1350	1313-1332	2.6E+05^{b}		6.3E+05^{a}		7.7E+05^{a}		*
18	Nonanal	124	19	6	1416	1376-1423	2.00E+05		3.2E+05		4.1E+05		NS
19	(E)-2-Octenal	2363	89	5	1462	1424-1467	2.30E+05		2.8E+05		3.5E+05		NS
20	Benzaldehyde	100	52	7	1573	1488-1585	2.1E+05^{c}		4.2E+05^{b}		6.7E+05^{a}		**
21	(E,E)-2,4-Decadienal	25152	84	5	1876	1763-1858	3.4E+05^{b}		5.7E+05^{a}		6.0E+05^{a}		**
	Total aldehydes						7.0E+06^{c}	29.8^{b}	1.1E+07^{b}	29.4^{b}	4.4E+07^{a}	43.7^{a}	***
Ketones													
22	2-Propanone	67	64	1	823	820-858	7.6E+05^{c}		1.1E+06^{b}		1.3E+06^{a}		**
23	2-Butanone	78	93	3	910	881-913	3.1E+04^{b}		4.1E+04^{b}		2.0E+05^{a}		***
24	2,3-Butanedione	431	3	8	980	975-1000	ND^{b}		ND^{b}		2.6E+05^{a}		***
25	2,3-Pentanedione	600	14	6	1065	973-1082	2.1E+04^{b}		4.9E+04^{b}		2.8E+05^{a}		***

Table 2 Volatile compounds identified by GC-MS-FID in *sués*, sautéed, and pan-fried onions *(Continued)*

26	2-Undecanone	112	12	9	1635	1570-1628	1.60E+05		1.7E+05		2.2E+05		NS
27	2-Tridecanone	593	8	8	1864	1783-1816	1.5E+05^{a}		1.2E+05^{a}		ND^{b}		***
	Total Ketones						1.1E+06^{c}	4.7	1.5E+06^{b}	3.9	2.3E+06^{a}	2.3	***
Hydrocarbons													
28	Pentane	109	66	0	500	500	3.60E+05		5.2E+05		5.7E+05		NS
29	Hexane	110	54	3	600	600	5.1E+04^{b}		1.3E+05^{a}		4.5E+04^{b}		**
30	Cyclopentane	287	92	3	634	/	8.20E+05		7.2E+05		8.3E+04		NS
31	1-Octene	111	66	0	859	822-892	4.9E+04^{b}		7.8E+04^{a}		8.8E+04^{a}		**
32	Decane	124	18	5	1000	1000	8.00E+04		1.3E+05		1.1E+05		NS
33	Dodecane	112	40	3	1193	1200	7.4E+04^{b}		4.9E+05^{a}		8.2E+04^{b}		***
34	Tetradecane	629	59	4	1400	1400	ND^{b}		8.4E+04^{a}		ND^{b}		***
	Total Hydrocarbons						1.40E+06	6.08^{a}	2.1E+06	5.7^{a}	9.7E+05	1.0^{b}	NS
Sulfur compounds													
35	Methanethiol	74	93	1	689	643-699	3.1E+04^{c}		6.0E+04^{b}		1.8E+05^{a}		***
36	Dimethyl sulfide	75	18	3	758	724-777	4.1E+04^{b}		3.5E+04^{b}		4.7E+05^{a}		***
37	1-Propanethiol	107	3	9	843	817-845	7.8E+04^{c}		1.5E+05^{b}		3.0E+05^{a}		***
38	*(Z)-1-Propenyl methyl sulfide*	52195	40	1	1003	1006	ND^{b}		ND^{b}		1.2E+05^{a}		***
39	*(E)-1-Propenyl methyl sulfide*	42848	6	6	1028	1006	5.5E+04^{b}		6.2E+04^{b}		1.3E+05^{a}		***
40	1-Pentanethiol	110	66	7	1055	1039-1055	4.4E+04^{c}		6.7E+04^{b}		8.0E+04^{a}		***
41	Dimethyl disulfide	624	92	0	1096	1057-1120	ND^{b}		ND^{b}		8.5E+05^{a}		***
42	2-Methylthiophene	554	14	3	1112	1078-1120	ND^{b}		ND^{b}		5.7E+04^{a}		***
43	3-Methyltiophene	616	44	4	1139	1093-1158	ND^{b}		5.0E+04^{b}		2.1E+05^{a}		***
44	*2,5-Dimethylthiophene*	638	2	8	1212	1187-1248	2.1E+05^{b}		3.0E+05^{b}		9.1E+05^{a}		***
45	*3,4-Dimethylthiophene*	632	15	5	1215	1240-1257	1.5E+05^{c}		3.0E+05^{b}		6.3E+05^{a}		***
46	Methyl propyl disulfide	2179	60	4	1257	1213-1243	1.3E+05^{b}		1.9E+05^{b}		5.3E+05^{a}		***
47	2,4-Dimethylthiophene	638	0	6	1278	1183-1264	1.5E+06^{c}		3.6E+06^{b}		1.1E+07^{a}		***
48	*(Z)-1-Propenyl methyl disulfide*	23838	18	8	1292	1245-1273	7.8E+05^{b}		1.5E+06^{b}		4.3E+06^{a}		***
49	*(E)-1-Propenyl methyl disulfide*	5905	47	5	1318	1268-1297	8.7E+05^{b}		2.4E+06^{b}		1.2E+07^{a}		***
50	Dipropyl disulfide	629	19	6	1411	1370-1396	3.4E+05^{a}		1.8E+05^{b}		1.1E+05^{b}		***
51	Dimethyl trisulfide	3658	80	8	1426	1370-1427	1.5E+05^{b}		4.9E+05^{b}		2.5E+06^{a}		***
52	*(Z)-1-Propenyl propyl disulfide*	23838	20	2	1451	1404-1407	7.3E+05^{a}		6.1E+05^{a}		4.0E+05^{b}		*
53	*(E)-1-Propenyl propyl disulfide*	5905	46	4	1474	1410-1447	1.20E+06		1.1E+06		1.3E+06		NS
54	Methional	3268	49	3	1490	1448-1479	ND^{b}		3.2E+05^{b}		4.1E+05^{a}		***

Table 2 Volatile compounds identified by GC-MS-FID in *sués*, sautéed, and pan-fried onions *(Continued)*

55	Methyl propyl trisulfide	17619	36	2	1586	1494-1521	4.20E+05		5.5E+05		6.8E+05		NS
56	Dipropyl trisulfide	6028	61	1	1737	1636-1738	2.5E+05^{a}		2.0E+05^{b}		1.5E+05^{c}		***
57	3,5-Diethyl-1,2,4-trithiolane	54644	28	9	1816	1744-1762	4.5E+05^{a}		3.2E+05^{ab}		2.5E+05^{b}		*
	Total sulfur compounds						7.4E+06^{b}	31.4^{b}	1.2E+07^{b}	32.2^{b}	3.8E+07^{a}	37.7^{a}	***
Furans													
58	2-Methylfuran	534	22	5	877	843-876	8.6E+04^{b}		1.0E+05^{ab}		1.4E+05^{a}		*
59	2,4-Dimethylfuran	3710	43	8	976	943-958	2.9E+06^{a}		3.68E+06^{a}		1.64E+05^{b}		***
60	2-Pentylfuran	3777	69	3	1242	1236-1243	4.1E+05^{ab}		6.4E+05^{a}		3.0E+05^{b}		**
61	Furfural	98	1	1	1494	1384-1493	1.3E+05^{c}		1.4E+06^{b}		2.0E+06^{a}		***
62	2-Acetylfuran	1192	62	7	1544	1475-1538	ND^{c}		1.2E+05^{b}		2.0E+05^{a}		***
63	5-Methylfurfural	620	2	0	1617	1542-1608	ND^{b}		3.4E+05^{a}		2.5E+05^{a}		***
64	2-Furanmethanol	98	0	0	1695	1636-1693	6.3E+05^{b}		1.1E+06^{a}		1.3E+06^{a}		*
	Total furans						4.16E+06^{b}	17.6	7.4E+06^{a}	19.7	4.4E+06^{b}	4.4	***
Pyrrole													
65	2-Acetylpyrrole	1072	83	9	2068	1935-2066	ND^{b}	0.0^{b}	ND^{b}	0.0^{b}	5.0E+06^{a}	0.5^{a}	***
Alcohol													
66	Ethanol	64	17	5	940	900-956	3.4E+05^{b}	1.4	4.6E+05^{b}	1.2	9.3E+05^{a}	0.9	*
	Total non identified compounds						1.78E+06^{c}	7.5^{ab}	2.68E+06^{b}	7.1^{b}	8.26E+06^{a}	8.3^{a}	***

Compounds in italics are those not confirmed by all means of identification and therefore considered as tentatively identified. Asterisks indicate differences between peak area values of onion preparations with a significance according to one-way analysis of variance *$p \leq 0.05$; **$p \leq 0.01$; ***$p \leq 0.001$. NS indicates no differences between peak area values of onion preparations according to one-way analysis of variance. Different letters (a, b, c) between columns indicate significant differences in the peak area values of onion preparations according to least significant difference test ($p \leq 0.05$)

produced in onions from the reaction between acetaldehyde and hydrogen sulfide [43].

Eighteen aldehydes were identified in the headspace of the three onion preparations: acetaldehyde, propanal, 2-methylpropanal, 2-propenal, butanal, 2-methylbutanal, 3-methylbutanal, pentanal, (E)-2-methyl-2-butenal, (E)-2-methyl-2-pentenal, heptanal, octanal, (E)-2-heptenal, nonanal, (E)-2-octenal, benzaldehyde, and (E,E)-2.4-decadienal. Their TPAs range from 29 to 30 % in the sautéed and *sué* onions samples up to 43.8 % in the pan-fried samples. Most of them are detected in higher amounts in the pan-fried onion headspace compared to *sué* and sautéed ones. Propanal was reported as being one of the most important aroma components in raw onions [44]. Actually, this compound is highly detected in the three onion preparations. It originates from the lachrymatory factor's decomposition. In raw onions, two molecules of propanal can further generate (E)-2-methyl-2-pentenal through aldol condensation [1]. A similar condensation of propanal and acetaldehyde can produce (E)-2-methyl-2-butenal which can be further reduced into 2-methylbutanal [44]. Acetaldehyde, propanal, (E)-2-propenal, 2-methylbutanal, 3-methylbutanal, 2-methylpropanal, and benzaldehyde are known to be Maillard reaction products, coming from Strecker degradation of corresponding amino acids [37], and except (E)-2-propenal and 2-methylpropanal, they all have been previously detected in heated onions [5, 6]. However, other chemical pathways can be involved in the formation of these compounds since acetaldehyde and 2-methylbutanal have also been reported in raw onions [6, 34]. Thermal degradation of lipids is another reaction which can produce numerous examples of the aldehydes detected in the present study (acetaldehyde, propanal, butanal, pentanal, hexanal, heptanal, (E)-2-heptenal, octanal, nonanal, (E)-2-octenal, and (E,E)-2,4-decadienal) through oxidation of linoleic and oleic acids which are present in sunflower oil [37, 44]. However, heptanal, (E)-2-octenal, and (E)-2-heptenal were also found in the volatile composition of onions heated without fat addition [5] which indicates that other reactions can produce aldehydes in onions and notably, endogenous enzymatic reactions [45].

Furans are compounds mainly detected in the onion preparations. Their peak areas represent up to 19.7 % of the TPA in the sautéed samples. Furfural, 5-methylfurfural, 2-furanmethanol, and 2-acetylfuran were previously reported in cooked onion. They are known to be involved in Maillard-type reactions of the sugars present in onions [46]. However, 2,4-dimethylfuran which is mainly detected in the three onion preparations and 2-methylfuran have never been reported in either cooked or raw onions and their origin remains unclear. 2-Pentylfuran, previously reported in processed onions [4, 7], could originate from thermal degradation of lipids [47] but could also originate from the thermal interaction of 2,4-decadienal with either cysteine or glutathione [37]. Hydrocarbons were also found in the volatile composition of the three cooked onion samples and, to our knowledge, only dodecane and tetradecane were previously detected in cooked onions [46]. Except cyclopentane, all hydrocarbons found in this study were detected in the volatile composition of oxidized sunflower oil so that oxidative degradation of the oil could be a possible source for these compounds [47, 48]. Hexane, dodecane, and tetradecane were detected in a significantly larger amount in the sautéed onion preparation.

2-Propanone, 2-butanone, 2,3-pentanedione, 2-undecanone, and 2-tridecanone identified in the present study were largely reported in the volatile composition of raw [6, 36, 42] or heated onions [6, 7]. Conversely, as far as we know, 2,3-butanedione has never been pointed out in the volatile composition of neither raw nor cooked onions, but this compound could originate from the Maillard reaction as well as 2,3-pentanedione [49]. Odd-chain ketones 2-undecanone and 2-tridecanone were previously reported in raw onions [6]. Although the function of these methyl ketones has not been reported in onions, these metabolites are present in tomato trichomes and confer insect resistance against a major agricultural pest, spiders [50–53]. The reactions by which methyl ketones are synthesized in plants have not been reported, but it has been hypothesized that these compounds could be synthesized either from beta-oxidation of fatty acids or by direct oxidation of hydrocarbons [54]. However, as far as we know, the origin of 2-propanone and 2-butanone is not clearly established in raw or cooked onions. The amounts of ketones detected in the headspace of the samples tend to increase in pan-fried preparation compared to the two other samples except for 2-undecanone which remains at equivalent amounts in each sample and for 2-tridecanone which is not detected in the pan-fried onions sample.

Acetic, propanoic, and hexanoic acids were identified in, at least, one of the onion preparations in amounts representing less than 1.5 % of TPA. To our knowledge, they have never been reported in raw onions but they were reported in the volatile composition of roasted onions [6]. This suggests that possible pathways for the formation of some of these compounds could be the thermal degradation of onion or sunflower oil [48]. Notably, hexanoic acid was mentioned to issue from thermal degradation of (E,E)-2,4-decadienal [44]. All acids were detected in significantly higher amounts in the headspace of the pan-fried onion preparation compared to the two other samples.

Ethanol was detected in low amounts, less than 2 % of TPA, in the headspace of the three onion preparations. This compound was previously identified in raw [6] and cooked onions [6, 13], but its origin in onion is unclear. It was detected in significantly higher amounts in the pan-fried onion preparation than in the other two.

2-Acetylpyrrole was detected in low amount (0.5 % of TPA) in the headspace of pan-fried onion samples solely. This compound occurs in various roasted products and issues from Maillard reaction [55]. It was previously detected in onions cooked by various processes [8, 13, 54].

A principal component analysis (Fig. 1), performed on peak areas of detected volatiles which undergo significant variation between at least two samples, illustrates the characteristics of each preparation regarding their chemical composition.

In accordance with statements reported above, the headspace of pan-fried onion sample is characterized by higher amounts of numerous compounds which is consistent for that preparation that combines high-temperature and long-time cooking and favors notably Maillard reaction and lipid oxidation. Conversely, the headspace of *sué* and sautéed preparations contains globally less compounds. However, the preparation of *sué* onion includes greater amounts of some compounds which are known to issue from enzymatic reactions: dipropyl disulfide, dipropyl trisulfide, 3,5-diethyl-1,2,4-trithiolane, and (Z)-1-propenyl propyl disulfide. This last compound is known to be an intermediate in dimethylthiophene formation [9]. Therefore, this result suggests that these three other sulfur compounds could be also involved as intermediates in further reactions. The headspace of the sautéed preparation is characterized by greater amounts of pentylfuran and heptanal coming from lipid oxidation, hexane, dodecane, and tetradecane, which were hypothesized to come from oxidation of lipids, as well as 2,4-dimethylfuran whose origin is unknown.

Comparison of the olfactometric profiles of *sué*, sautéed, and pan-fried onions

The laboratory-developed software used to acquire olfactometric data succeeds in permitting a very intuitive categorization and rating of odors perceived by judges. The results of the olfactometric study are presented in Table 3.

In the *sué* and sautéed onion samples, 50 and 53 odorant zones were significantly detected whereas 65 were perceived in the pan-fried samples. A total of 71 different odorant zones were listed. Most of them, i.e., 42 zones, were significantly perceived in each preparation which means that a major part of the odorants is common to the three samples. Hence, flavor differences resulting from these three modes of cooking are due to a minor part of the present odorants.

Among the 42 zones commonly perceived in the *sué*, sautéed, and pan-fried preparations, 12 were perceived

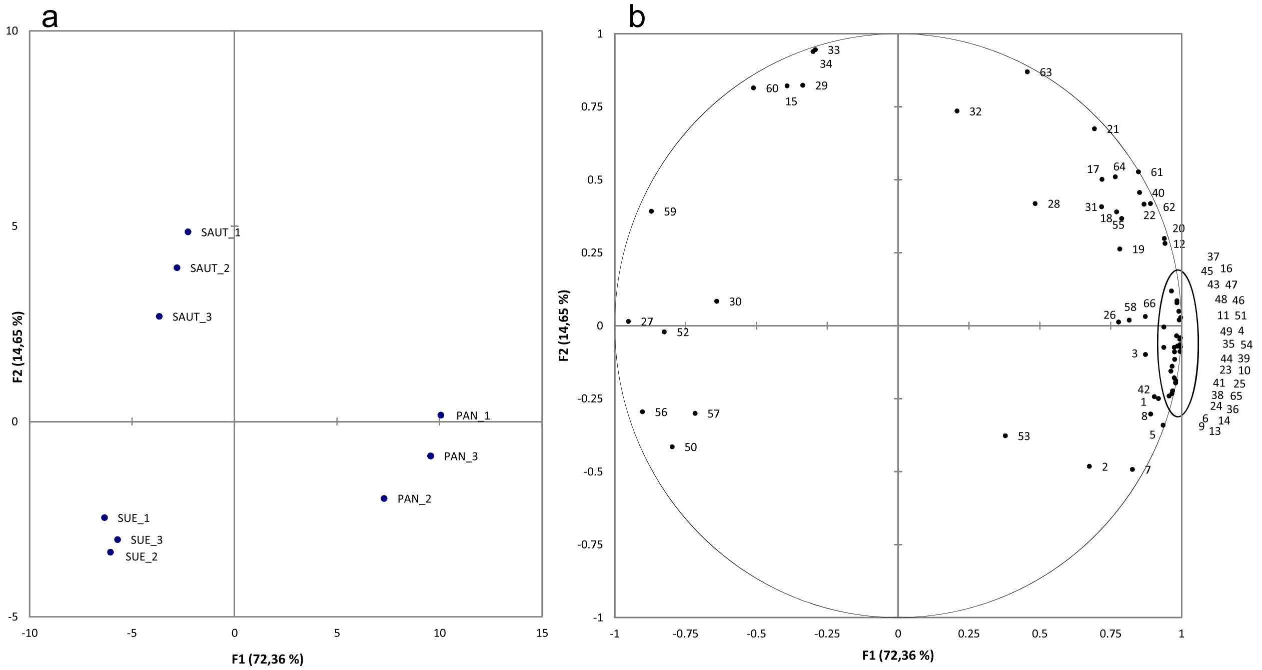

Fig. 1 Principal component analysis performed on the volatile compounds identified in *sué*, sautéed, and pan-fried onion. **a**- Score plot from onion preparations. **b**- Loading plot for volatile compounds (numbers correspond to those associated with compounds in Table 2

Table 3 Olfactometric profiles of *sués,* sautéed, and pan-fried onions

LRI experiment	Compound	CAS number			Poles and descriptors (in brackets) most cited by judges	Identification		*Sué* onion		Sauteed onion		Pan-fried onion		Significance
						LRI in literature	Compound odor description in the literature	Detection	Average score	Detection	Average score	Detection	Average score	
545	**Hydrogen sulfide**	7784	6	4	Animal (feet), sulfurous	480-560	Rotten egg (a)	1	0.3	3	0.3	3	1.0	NS
701	**Methanethiol**	74	93	1	Animal (feet), sulfurous (cabbage)	643-699	Sulfurous (a)	5	1.9	5	2.5	7	3.8	NS
	Acetaldehyde	75	7	0		677-744	Etherial (a)							
769	**Dimethyl sulfide**	75	18	3	No consensus	724-777	Sulfurous (a)	0	0.0^{b}	0	0.0^{b}	4	1.3^{a}	**
802	**Propanal**	123	38	6	Chemical, acid	769-828	Etherial (a)	2	0.4	1	0.1	3	1.3	NS
824	**2-Methylpropanal**	78	84	2	No consensus	820-858	Spicy (a)	0	0.0^{b}	1	0.10^{b}	5	2.8^{a}	***
844	**1-Propanethiol**	107	3	9	Sulfurous	817-845	Alliaceous (a)	7	4.4	6	4.3	7	4.6	NS
895	No peak				Sulfurous			3	1.0	1	0.3	5	2.0	NS
926	**3-Methylbutanal**	590	86	3	Animal (feet), malty	902-949	Aldehydic (a)	2	0.6^{b}	6	3.7^{a}	7	5.7^{a}	***
936	*Propenthiol*	925	89	3	Sulfurous, pyrogenic (broth)	895-960	Raw Welsh onion (b)	8	5.3	8	5.5	8	5.9	NS
	and/or Propylene sulfide	1072	43	1		875-915								
979	**2,3-Butanedione**	431	3	8	Aldehydic (butter), sweet	975-1000	Buttery (a)	4	1.7	5	3.1	6	4.0	NS
1001	*(Z)-1-Propenyl methyl sulfide*	52195	40	1	Sulfurous, pyrogenic, unknown	1006	Garlic (a)	0	0.0^{b}	0	0.0^{b}	4	1.8^{a}	*
1021	*(E)-1-Propenyl methyl sulfide*	42848	6	6	Sulfurous, pyrogenic	1006	Garlic (a)	1	0.2^{b}	4	1.0^{ab}	4	2.0^{a}	*
1051	*1-Pentanethiol*	110	66	7	Sulfurous, animal	1039-1055	Sulfurous (a)	4	1.6	5	1.9	3	1.5	NS
1060	**2,3-Pentanedione**	600	14	6	Aldehydic (butter), sweet	973-1082	Buttery (a)	0	0.0^{b}	0	0.0^{b}	4	1.8^{a}	**
1096	**Hexanal**	66	25	1	Herbal, pyrogenic (caramel)	1067-1099	Green (a)	2	0.7	3	1.4	3	1.1	NS
1110	**Dimethyl disulfide**	624	92	0	Sulfurous	1057-1120	Sulfurous (a)	3	0.7^{b}	7	3.1^{a}	6	2.2^{a}	**
	2-Methylthiophene	554	14	3		1078-1120	Sulfurous (a)							
1126	*(Z)-1-Propenyl propyl sulfide*	33922	70	2	Sulfurous		Garlic (a)	4	1.6	5	2.0	6	2.6	NS
	and/or (E)-1-Propenyl propyl sulfide	37981	34	3										
1139	**3-Methylthiophene**	616	44	4	Sulfurous	1093-1158		5	2.4^{ab}	3	1.4^{b}	6	3.9^{a}	*
1152	No peak				Sulfurous			5	2.5	6	2.9	5	3.2	NS
1169	**Diallyl sulfide**	592	88	1	Sulfurous	1118-1170	Sulfurous (a)	4	1.4	3	1.5	5	2.7	NS
1195	Unknown peak				Sulfurous (gas)			3	1.5	3	1.5	1	0.4	NS

Table 3 Olfactometric profiles of *sués,* sautéed, and pan-fried onions *(Continued)*

1221	*2,5-Dimethylthiophene*	638	2	8	Sulfurous, pyrogenic	1187-1248	Sulfurous (a)	4	1.5	5	1.2	5	1.7	NS
	3,4-Dimethylthiophene	632	15	5		1240-1257	Roasted onion (a)							
1237	Unknown peak				Sulfurous			0	0.0^{b}	0	0.0^{b}	3	1.6^{a}	*
1248	**Methyl propyl disulfide**	2179	60	4	Sulfurous	1213-1243	Alliaceous (a)	0	0.0^{b}	4	1.3^{a}	4	1.3^{a}	*
1261	Unknown peak				Sulfurous			0	0.0	5	1.5	3	1.7	NS
1281	**2,4-Dimethylthiophene**	638	0	6	Sulfurous	1183-1264	Boiled onion-like (c)	7	3.8	7	5.3	8	5.6	NS
1315	**1-Octen-3-one**	4312	99	6	Woody (mushroom)	1298-1323	Earthy (a)	8	6.0	8	6.2	8	6.5	NS
1331	Unknown peak				Sulfurous			6	2.8^{a}	1	0.6^{b}	3	1.7^{ab}	***
1346	Unknown ((E)-2-heptenal trail)				Sulfurous			3	1.6^{b}	4	2.0^{b}	7	4.3^{a}	***
1368	**2,6-Dimethylpyrazine**	108	50	9	Pyrogenic (roasted)	1300-1360	Chocolate (a)	5	1.9^{b}	7	4.8^{a}	6	4.1^{a}	**
	Ethylpyrazine	13925	0	3		1334-1353	Nutty (a)							
1387	No peak				Sulfurous, pyrogenic			3	1.1	1	0.7	1	0.8	NS
1398	No peak				Sulfurous			3	0.7	4	1.7	6	2.5	NS
1414	**Dipropyl disulfide**	629	19	6	Sulfurous	1370-1396	Alliaceous (a)	8	5.6	8	5.1	8	5.7	NS
	Dimethyl trisulfide	3658	80	8		1370-1427	Alliaceous (a)							
1423	Unknown peak				Chemical, sulfurous, pyrogenic			8	5.3	8	4.5	8	5.1	NS
1444	Unknown peak				Sulfurous			4	2.2	5	2.6	5	1.8	NS
1449	*(Z) 1-Propenyl propyl disulfide*	23838	20	2	Sulfurous	1404-1407	Baked Welsh onion-like (b)	2	1.4	1	0.6	4	1.8	NS
1462	*(E) 1-Propenyl propyl disulfide*	5905	46	4	Sulfurous	1410-1447	Raw onion-like (b)	6	2.4	7	3.2	5	2.2	NS
	and/or allyl propyl disulfide	2179	59	1		1386-1474	Sulfurous (a)							
1479	**Methional**	3268	49	3	Sulfurous (cooked potatoes)	1448-1479	Vegetable (a)	8	6.4	8	6.6	7	6.0	NS
1487	**Furfural**	98	1	1	Pyrogenic (cooked vegetables), woody	1384-1493	Bready (a)	1	0.1^{b}	2	0.6^{b}	5	2.6^{a}	***
1507	*Diallyl disulfide*	2179	57	9	Sulfurous, pyrogenic	1463-1256	Alliaceous (a)	0	0.0^{b}	0	0.0^{b}	3	1.5^{a}	**
1533	**2-Acetylfuran**	1192	62	7	Sulfurous, pyrogenic	1475-1538	Balsamic (a)	3	1.6^{b}	3	0.8^{b}	8	4.9^{a}	***
1545	*No peak*				Sulfurous			7	3.8	3	1.7	6	3.8	NS
1553	Unknown peak (acide propanoic trail)				Sulfurous			4	2.4	3	1.3	4	2.5	NS
1558	Unknown peak (benzaldehyde trail)				Sulfurous			5	3.4	4	2.0	5	2.6	NS
1562	No peak				Sulfurous, pyrogenic			6	3.8	4	2.4	5	2.2	NS
1574	Unknown peak				Sulfurous			8	5.4^{a}	8	6.1^{a}	7	3.3^{b}	**

Table 3 Olfactometric profiles of *sués,* sautéed, and pan-fried onions *(Continued)*

1591	No peak	17619	36	2	Sulfurous	1494-1521	Sulfurous (a)	6	4.7	7	5.1	5	3.0	NS
1599	Unknown peak (5-methylfurfural trail)				Sulfurous			8	5.2	7	6.0	7	5.1	NS
1621	**Dimethyl sulfoxide**	67	68	5	Sulfurous	1560-1603	Alliaceous (a)	8	5.7	7	2.9	7	5.6	NS
1636	No peak				Sulfurous			5	3.3	6	3.7	5	3.9	NS
1651	*Allyl methyl trisulfide*	34135	85	8	Sulfurous	1592-1605	Sulfurous (a)	7	5.1	8	5.6	8	5.6	NS
	and/or (Z)-Propenyl methyl trisulfide	33368	80	8		1605								
	and/or (E)-Propenyl methyl trisulfide	23838	25	7										
1675	**Benzeneacetaldehyde**	122	78	1	Flower, pyrogenic (roasted), sulfurous, aldehydic	1638-1684	Green (a)	6	4.1	6	2.6	4	1.8	NS
	Butyrolactone	96	48	0		1609-1672	Creamy (a)							
	2-Furanmethanol	98	0	0		1636-1693	Bready (a)							
1699	*3 -Thiophenecarboxaldehyde*	498	62	4	Sulfurous	1666-1693		8	5.6	8	5.5	8	5.6	NS
	Dipropyl trisulfide	6028	61	1		1636-1738	Sulfurous (a)							
1735	**2-Thiophencarboxaldehyde**	98	3	3	Sulfurous, aldehydic	1655- 1734		6	4.5	5	2.6	6	2.8	NS
1752	No peak				Sulfurous			1	0.8	4	2.1	5	2.9	NS
1778	No peak	23838	27	9	Sulfurous (raw onion)	1749-1795		5	2.0	6	3.1	1	0.9	NS
1796	*(Z)-Propenyl propyl trisulfide*	23838	26	8	Chemical, pyrogenic, sulfurous	1728	Onion-like (c)	4	2.1	3	1.3	3	1.1	NS
	and/or (E)-Propenyl propyl trisulfide	23838	27	9		1750	Onion-like (c)							
	and/or Allyl propyl trisulfide	33922	73	5		1699	Sulfurous (a)							
1815	Unknown peak				Sulfurous	1762-1785		0	0.0^{b}	0	0.0^{b}	3	1.4^{a}	*
1854	*Diallyl trisulfide*	2050	87	5	Sulfurous, pyrogenic	1775-1789	sulfurous (a)	7	5.1^{b}	8	5.99^{a}	8	5.87^{a}	***
	Dipropenyl trisulfide (isomer)													
1858	**(E,E) 2,4-Decadienal**	25152	84	5	Sulfurous, unknown	1763-1858	Fatty (a)	6	3.0	5	3.4	6	3.9	NS
1884	Unknown				Pyrogenic, unknown			3	1.5^{a}	0	0.0^{b}	5	2.1^{a}	*
1954	No peak				Sulfurous (sauteed/ pan fried), pyrogenic			3	1.3	2	1.0	2	0.8	NS
1975	Unknown peak				Sulfurous			0	0.0	4	1.1	3	1.1	NS
2043	**2-Acetylpyrrole**	1072	83	9	Other, sweet, floral	1935-2066	Musty (a)	4	1.4	3	1.4	6	2.4	NS
2071	Unknown peak				Other, pyrogenic			5	3.0	5	1.4	6	2.8	NS
2100	No peak				Pyrogenic (caramel)			6	3.7^{b}	7	5.0^{a}	7	6.0^{a}	***
2115	No peak				Pyrogenic (caramel)			6	2.9	3	2.1	3	2.3	NS
2178	No peak				Pyrogenic			1	1.2	3	0.8	1	0.9	NS

Table 3 Olfactometric profiles of *sués,* sautéed, and pan-fried onions *(Continued)*

2216	No peak	Pyrogenic (roasted), sulfurous	3	1.1	0	0.0	1	0.5	NS
2241	No peak	Sulfurous	0	0.0^b	0	0.0^b	3	0.8^a	*
2255	No peak	Sulfurous, pyrogenic	0	0.0^b	0	0.0^b	3	1.0^a	*

Compounds in bold are those for which identification is considered as achieved (based on LRI, MS, odor, and standard). Compounds in italics are those not confirmed by all means of identification and therefore considered as tentatively identified. The gap that can be observed between LRI of some compounds and LRI of the corresponding odorant zones is due to the difference of temperature between the transfer line (200 °C) and the capillary leading to the MS detector (equal to oven temperature). Asterisks indicate differences between intensity score of onion juices with significance according to two-way analysis of variance $*p \leq 0.1$; $**p \leq 0.05$; $***p \leq 0.01$. NS indicates no differences between intensity scores of onions juices according to two-way analysis of variance. Different letters (a, b) between columns indicate significant differences in the intensity scores of onions juices according to least significant difference test ($p \leq 0.1$). For each odorant zone, the non-consensual descriptors belonging to a same pole were grouped by the name of the pole. Odor description: (a) [61], (b) [7, 8], (c) [13] by 7 or 8 judges out of 8 in each sample. 1-Octen-3-one (linear retention index (LRI)—1315) was unanimously detected and described as mushroom. However, a majority of these zones were described by judges as sulfurous and 8 were actually associated with sulfur compounds (LRI 936—propenthiol and propylene sufide; LRI 1281—2,4-dimethylthiophene; LRI 1414—dipropyl disulfide and dimethyl trisulfide; LRI 1479—methional; LRI 1621—dimethyl sulfoxide; LRI 1651—allyl methyl trisulfide and/or (Z and/or E)-1-propenyl methyl trisulfide; LRI 1699—3-thiophenecarboxaldehyde and dipropyl trisulfide; LRI 1854—diallyl trisulfide and/or dipropenyl trisulfide). These compounds are supposed to contribute largely to the aroma of the studied samples. Propyl- and propenyl-containing di- and trisulfides were previously reported to contribute to the flavor of cooked onions [6, 8]. These profiles seem to confirm the involvement of 2,4-dimethylthiophene in the aroma of "fried" cooked onions [9], but as mentioned previously [12], the odorant note associated with this compound was not described as such. Contrary to the other compounds found in the olfactometric profile of the three onion preparations, the terms given by judges to describe the odor of sulfurous compounds were not consensual but they all belong to the sulfurous pole. Therefore, only the general term "sulfurous" corresponding to this pole was retained. Other odorant zones perceived in each product were also mainly described as sulfurous and those which have been identified were associated with thiols (LRI 701—methanethiol; LRI 844—1-propanethiol; LRI 1051—1-pentanethiol), sulfides (LRI 1126—(Z) or (E)-1-propenyl propyl sulfide; LRI 1169—diallyl sulfide), disulfides (LRI 1462—allyl or (E)-1-propenyl propyl disulfide, LRI 1110—dimethyl disulfide), trisulfides (LRI 1796—(E) and/or (Z)-1-propenyl propyl trisulfide and/or allyl propyl trisulfide), and thiophenes (LRI 1110—2-methylthiophene; LRI 1139—3-methylthiophene; LRI 1221—2,5- and 3,4-dimethylthiophene; LRI 1735—2-thiophenecarboxaldehyde). Some compounds perceived in all products also bring non-sulfurous notes characterized by descriptors belonging to the aldehydic pole (LRI 979—2,3-butanedione; LRI 1675—butyrolactone; LRI 1858—2,4-decadienal) and to the pyrogenic pole (LRI 1675—2-furanemethanol; LRI 1368—2,6-dimethylpyrazine and ethylpyrazine; LRI 1533—2-acetylfuran). Additional odorant zones detected in the three samples could not be associated with a compound. They were also mainly described by descriptors from the sulfurous and pyrogenic poles (LRI 1152, 1346, 1398, 1444, 1545, 1553, 1558, 1562, 1591, 1636, 2071, 2100, and 2115). The prevalence of sulfurous detections in the olfactometric profiles of the cooked onions can be explained by the preponderance of sulfur compounds in onion but particularly by the very low detection thresholds of these compounds that could be within thousandths of a part per billion [56]. This could also explain that many odorant zones described as sulfurous remained unknown or not associated with any peak since compounds are probably present in trace amounts. Very little bibliography deals with the aromatic profile of cooked onions. To our knowledge, studies dealing with olfactometry analysis were performed on onions cooked without fat which can logically explain that compounds identified as coming from thermal degradation of lipids (acetaldehyde, 1-octen-3-one, 2,4-decadienal) were not previously listed [8, 13]. Furthermore, in these two previous studies, olfactometry was performed with an unknown number of judges, which can explain that only half the odorant zones that were perceived in each of the three present preparations were previously detected.

Beyond the single detection of the odorant compounds, the use of the laboratory-developed software enables a rating of the intensities perceived by judges for each odorant zone on a continuous scale. An analysis of variance was carried out on the intensity scores obtained for each odor event in the three samples. It reveals that 22 odorant zones out of the 71 detected were perceived as significantly different in at least one of the three onion preparations (p value <0.1).

The olfactometric profile of the *sué* onion sample singles out by the lowest number of odorant zones detected associated with low intensities scores. This result is in accordance with the result of ANOVA performed on major compounds' FID peak areas presented above.

Conversely, the headspace of the pan-fried onion sample is characterized by a higher number of odorant zones detected and also by significantly higher intensity scores mainly for compounds that originate from Maillard reaction bringing notes belonging to the pyrogenic pole (LRI 824—2-methylpropanal; LRI 1139—3-methylthiophene; LRI 1487—furfural; LRI 1533—2-acetylfuran). The higher detection of these two latter compounds is consistent with the bibliography that identify furanic compounds as important contributors to the characteristic odor of fried products [57]. These compounds have low thresholds and provide pleasant odor characteristics, such as cocoa, butter, or fruity [58]. The present results are in accordance with previous observations of a reduced pungency of onion through sweet notes with cooking [44]. Some odorant zones are only detected in the headspace of the pan-fried samples and were related either to sulfur compounds (LRI 769—dimethyl sulfide; LRI 1001—(Z)-1-propenyl methyl sulfide) or to compounds generated by Maillard reaction (LRI 1060—2,3-pentanedione). These results are also consistent with those of ANOVA performed on FID peak areas presented above.

Intermediately, the headspace of the sautéed onion sample is qualitatively close to that of the *sué* sample with similar odorant zones detected. However, one third of them were significantly perceived as more intense. Among them, 3-methylbutanal (LRI 928) was associated with malted and animal odorant notes. The others are both related to sulfur and Maillard compounds and are mainly associated with sulfurous and pyrogenic descriptors, respectively. Particularly, the presence of pyrazines at LRI 1368 described as "roasted" [59, 60] and the unidentified odorant zone at LRI 2100 described as caramel seems to contribute to the specific aroma of the sautéed onions.

Handling of data was simplified by the use of this software that records complete odor informations for odor events, i.e., elution time, duration, intensity, and descriptors, and provides computerized and ready-to-process data directly after gas chromatography-olfactometry (GC-O) sessions. Individual aromagrams can be automatically combined for each sample either into detection frequency or average intensity aromagrams. These aroma profiles were obtained from judges through an intuitive and rapid demarch which increase result accuracy. Judges can thus properly transmit characteristics of close odor events. As an example, judges succeed to characterize properly the close odorant areas corresponding to methional (LRI 1479) and furfural (LRI 1487).

Besides, the fact that, beyond simple detection, this software can record intensity score from a continuous scale, allows going further in the comparison of resembling aroma products such as these three traditional onions preparations. ANOVA performed on intensity scores highlights for some compounds, differences that were not perceptible by comparison of their detection frequency. As an example, two unidentified odor areas, associated with sulfurous (LRI 1574) and caramel (LRI 2100) descriptors, respectively, were similarly perceived by six to eight judges in the three samples. ANOVA performed on their intensity scores underlined significative differences since the less intense odor area has an intensity average between 3.3 and 3.7 while the others were between 5.0 and 6.1 which pointed out that the compound associated with the first cited odorant zone (LRI 1574) is likely to more impact the sulfur characteristic aroma of the sué and sautéed onions than that of pan-fried onion. Conversely, the unidentified compound associated with the second cited odorant zone (LRI 2100) can bring a caramel note in the pan-fried onion aroma in a greater manner than in the other two.

Conclusions

This study results in the aromatic characterization of onions prepared through three traditional modes of cooking, i.e., *sué,* sautéed, and pan-fried. The analysis of samples by gas chromatography coupled with flame ionization detection and mass spectrometry allows the identification of 66 major compounds. Among them, sulfur compounds, aldehydes, and furanic compounds were the most represented according to their FID peak areas. The headspace of *sué* and sautéed preparations contains globally fewer compounds than the headspace of the pan-fried samples. The *sué* sample contains greater amounts of some sulfur compounds coming from enzymatic reactions whereas the headspace of the sautéed preparation is characterized by greater amounts of some compounds hypothesized to come from lipid oxidation. Additionally, the headspace of the pan-fried onion sample is characterized by higher amounts of many compounds which are consistent for that preparation which combines high temperature with long cooking time and favors notably Maillard reaction and lipid oxidation.

The olfactometric approach completes the characterization of these three samples of cooked onions revealing the contribution of minor compounds to their specific aromas. The use of innovative laboratory-designed software enables an intuitive characterization and precise rating of the odorants present in these products and thus allows a statistical comparison of their aromatic profiles. In accordance with the chromatographic results, the *sué* onions single out by a weak number of detected odorant zones associated with low intensity scores. Conversely, the pan-fried onions are characterized by more odorant zones detected associated with higher intensities and notably by an enhanced perception of some Maillard compounds. The aromatic profile of sautéed onion is qualitatively close to that of *sué* onion but is associated with more important intensities. Particularly, the presence of pyrazines could contribute to the specific aroma of the sautéed onions. This knowledge can be notably capitalized by food industry to create or enhance culinary notes in food products.

The assessment of olfactometric profiles obtained for onions prepared by three different modes of cooking and the comprehension of such fine nuances in aroma could not have been performed without the precision and the accuracy of data recorded by the laboratory-developed software coupled with the statistical processing of intensity scores. Indeed, this innovative tool allows a rapid and efficient data transmission and recording of perceptions through an intuitive wheel aroma interface. It solves most bias found in current GC-O data acquisition methods (Table 1) and notably those inherent to the oral transmission of judges' impressions. This device is a valuable tool to investigate products with complex aroma, identify target compounds involved in specific aroma notes, and follow the evolution of aroma profile of a product during its fabrication process or storage. It gives insights to food industry to understand, reproduce, or enhance culinary notes.

Methods

Onion sample preparation

Fresh, raw onions were peeled and chopped into 3–5 mm cubes and then cooked in accordance with definitions of the *French Larousse Gastronomique* to obtain *sué*, sautéed, and pan-fried onions.

Sué onion preparation: 30 g of sunflower oil were added to a saucepan heated to 100 °C and 1 kg of onions was then added. The onions were regularly stirred for 25 min. Cooking was stopped when the onions were translucent.

Sautéed onion preparation: 30 g of sunflower oil was heated to 155 °C in a pan. Then, 1 kg of onions was added and was evenly sautéed for 10 min. Cooking was stopped when onions had a homogeneous caramelized appearance.

Pan-fried onion preparation: 30 g of sunflower oil was heated to 130 °C in a pan. Then, 500 g of onions were added and were evenly sautéed for 18 min. Cooking was stopped when onions had a shiny appearance and some of them were burnt.

Each of the three preparations were pounded and pressed into onion juice. Aliquots of 7 mL of juice were put into 20-mL glass vials, hermetically closed with a metal/Teflon cap. These samples were stored at −80 °C until analysis.

Extraction of volatile compounds

Headspace solid phase micro-extraction (HS-SPME) was used to extract volatile compounds of onion juices samples. Vials were incubated at 45 °C for 45 min, and volatile compounds were then extracted on a CAR/PDMS SPME fiber (10 mm long, 85 μm film thickness; Supelco, Bellefonte, PA, USA) placed in the headspace of the vial for 10 min.

Chromatographic conditions

HS-SPME extracts of onion juices were analyzed by gas chromatography (GC; Agilent Technologies 7890N, Wilmington, DE, USA) coupled with a quadripolar mass spectrometer (MS; Agilent Technologies, 5973 Network, Wilmington, DE, USA), a flame ionization detector (FID), and an olfactometric port. Volatile compounds were desorbed into the injection port of the chromatograph (temperature 260 °C; splitless mode for 5 min) and separated on a DB-Wax column (length 30 m, internal diameter 0.25 mm, film thickness 0.5 μm). Helium was used as carrier gas at constant pressure (124 kPa). The oven temperature was programmed from 40 (0 min) to 50 °C at 5 °C·min^{-1}, next from 50 to 120 °C (2 min) at 10 °C·min^{-1}, then from 120 to 210 °C at 10 °C·min^{-1}, and finally from 210 to 240 °C (10 min) at 25 °C·min^{-1}. Effluent from the end of the GC column was split 1:3 between the MS, the FID, and the olfactometric port (250 °C, air/H2 flow 450/40 mL·min^{-1}). Peak areas were integrated using MSD Chemstation software (Agilent Technologies). Mass spectra were recorded in electron impact mode (70 eV) between a mass range of 33 to 300 *m*/*z* at a scan rate of 2.7 scan·s^{-1}.

Olfactometric conditions

GC effluent was carried to the sniffing port using a deactivated and uncoated fused silica capillary column, heated to 200 °C. The GC sniffing port is equipped with a nose glass funnel where assessor puts his nose. The olfactometric port was supplied with humidified air to prevent dehydration of the nasal mucosa.

Olfactometric data were recorded in real time with laboratory-designed software that records the following parameters synchronously the GC-MS analysis: times of perception of an odor, intensity, and associated descriptor. Descriptors were generated from literature on aroma compounds found in raw and cooked onions [8, 13, 61, 62] and from previous sniffing sessions performed by two expert judges on each of the three onion preparations. They were presented on a dedicated wheel aroma especially designed for the study of cooked onions samples (Fig. 2).

The wheel is structured in 17 poles associated with general odor families written in capital letters. These poles can be divided in more numerous sections associated with precise descriptors. Colors were also associated with poles to help judges rapidly find terms corresponding to the odors perceived. Judges were trained in aroma recognition and in the use of an intensity scale. Terms were explained to the judges, and the panel was trained to locate each pole and descriptor on the wheel according to the Table 4.

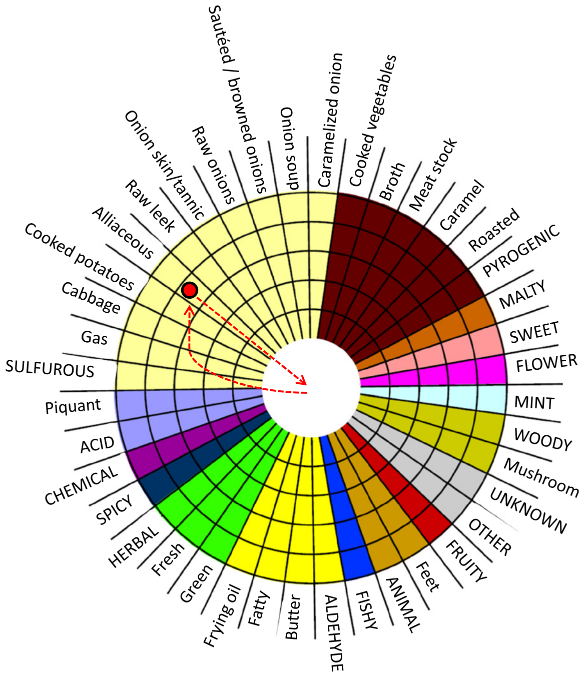

Fig. 2 Aroma wheel used for the olfactometric analysis of sué, sautéed, and pan-fried onion preparations

Table 4 Terms used to describe odors perceived during olfactometric analysis of onions and examples associated

Odor families	Terms	Examples associated
Fruity		
Animal		
	Feet	
Fishy		Fish, ammoniacal
Aldehyde		
	Butter	Fresh butter, melted butter
	Fatty	Animal fat, margarine, lard
	Frying oil	Oily, french fries
Herbal		
	Green	Green vegetables
	Fresh	Cutted grass
Spicy		Clove, pepper, curry
Chemical		Pharmaceutical, medicine, solvent, alcohol
Acid		
	Piquant	vinegar
Sulfurous		
	Gas	Natural gas
	Cabbage	
	Cooked potatoes	Boiled potatoes, purée
	Alliaceous	Garlic, shallot, chive, ciboule
	Raw leek	
	Onion skin/tannic	
	Raw onions	
	Sautéed/browned onions	
	Onion soup	
	Caramelized onion	
Pyrogenic		
	Cooked vegetables	
	Broth	Soup
	Meat stock	
	Caramel	Melted sugar, salted butter caramel
	Roasted	Torrefied coffee, toasted bread, grilled meat
Malty		Chocolate, beer, basmati rice
Sweet		Candy, vanilla
Flower		
Mint		Chewing gum with menthol, anise
Woody		
	Mushroom	Wet cellars, earthy, musty
Unknown		
Others		

Ethical committee approval was not required for this study; however, human sensory analyses were conducted following the spirit of the Helsinki Declaration, and informed consent was obtained from all panelists.

Each judge had prior experience in GC-O. Eight judges were involved in the olfactometric analysis of each sample. During olfactometric sessions, they were encouraged to describe each odor perceived as precisely as possible using terms proposed on the wheel. If the odor perceived did not correspond to any descriptor, they were invited to describe, if possible, the odor by the name of the pole corresponding to the general odor family. If the odor perceived could be neither related to a descriptor nor to a general family, judges could use the "Unknown" section or the "Other" section. Only in this latter case, the description of the odor was externally recorded.

Judges were asked to signal the perception of an odor by directing the mouse pointer toward the section of the wheel corresponding to the adequate odor term or pole. They were also asked to score the intensity of the perceived odor on a 0–10 intensity scale. The scale was represented by the radius of the wheel; the center of the wheel stands for the zero value and the edge of the wheel for the 10 (maximum) values. The judges scored the odor by clicking in the corresponding scale level. When an odor was no longer perceived, judges were asked to direct the pointer of the mouse back to the wheel center.

Odorant compound identification

Odorant compounds were identified by comparison of their mass spectra with those of a reference database (Wiley 6.0 and internal laboratory database). Linear retention indices (LRI) of detected compounds were calculated by means of n-alkane injections (C6 to C32) and compared with those of standards injected in the same conditions and with those found in the literature. Descriptors given for each detected compound were also compared with those found in the literature.

Data processing and statistical analyses

FID peak areas One way analysis of variance (ANOVA) was performed on FID peak areas. Least significant difference (LSD) multiple comparison tests were then performed with a 95 % confidence level. A principal component analysis was conducted on peak areas significantly different between at least two products as highlighted by ANOVA.

Olfactometric data Odorant zones detected by at least three judges in at least one sample were taken into account [23]. Two-way analysis of variance and LSD multiple comparison tests (90 % confidence level) were performed on intensity scores in order to highlight the differences between the olfactometric profiles of each type of onion preparations.

Xlstat software (version 2011.2.08, Addinsoft) was used to conduct these statistical analyses.

Abbreviations

ANOVA: analysis of variance; FID: flame ionization detection; GC: gas chromatography; HS-SPME: headspace solid phase microextraction; LRI: linear retention indices; LSD: least significant difference; MS: mass spectrometry; O: olfactometry; TPA: total peak area.

Competing interest

The authors declare that they have no competing interests.

Authors' contributions

AV conceived of the study, analyze the results, and write the manuscript. SLR participated in analyzing the results and writing the manuscript. CF carried out the experiments and participated in analyzing the results. FG participated in the realization of the olfactometric software. HF and SB designed procedures for optimal onion preparations. CP conceived of the study and revised the manuscript. All authors approved the final document.

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